

C Evaluation of laboratory blank contamination with validation of data from samples associated with contaminated blanks using the following guidance.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. No positive sample results should be reported unless the concentration of the compound in the sample exceeds five times the amount (in any blank). In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value. Specific actions are as follows:

1. If a compound is found in a blank, but not found in the sample, no action is taken.
2. If a blank has a positive result for an analyte, qualify associated sample data as follow:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag the sample result as a non-detect (UL). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detected at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5 x criteria, such that a comparison of the total contamination is actually made.

3. Evaluation of calibration and tuning information to determine compliance with specifications contained within the individual methodologies.
4. The criteria for evaluation of compound quantitation/transcription errors is based on guidance from Region II Sop No. HW-6: CLP Organics Data Review and Preliminary Review. Evaluation of compound quantitation will be performed by recalculation of sample values from the raw data for at least two positive values per data package. If problems are found, additional data calculations will be checked.

### 8.2.2 Sample Specific Criteria

All of the data packages from each laboratory for each analysis type will be reviewed for the following sample specific criteria, as appropriate to the analytical method:

- C Holding Times
- C Matrix Spike/Matrix Spike Duplicate Analysis (MS/duplicate for inorganics)
- C Surrogate Recovery (Organics)
- C Laboratory and Field Blank Results
- C Target Compound Identification (Organics)
- C Laboratory Replicate Analysis

In addition to the sample specific criteria listed above, the reviewer will investigate the effect of any systematic problems noted in the review of the laboratory performance

criteria and of any problems noted by the laboratory in the Case Narrative and qualify data where appropriate.

The Region II SOPs will be used for validation of the sample specific criteria for the volatile organics, semivolatile organics, pesticide/PCBs, metals and cyanide, and PCDDs/PCDFs analyses as discussed in Section 8.2 above. Validation and qualification of the sample specific criteria for the chlorinated herbicides analyses, total extractable petroleum hydrocarbons analyses, radiochemical analyses and analyses of other non-SW-846 analyses are specified below.

#### **8.2.2.1 General**

Homogenized field duplicates will be evaluated against the following criteria. Where both the sample and duplicate values are greater than 5 times the SQL, acceptable sampling and analytical precision is indicated by an RPD for the two field duplicate results of less than or equal to 70 percent. Where one or both analytes of the field duplicate pair are less than 5 times the SQL, satisfactory precision is indicated if the field duplicate results agree within 2.5 times the SQL. If the above criteria are not met for an analyte, qualify all associated sample data for that analyte as estimated (U).

All sample results reported as detected below the reported sample quantitation limit will be qualified as a non-detect at the SQL.

#### **8.2.2.2 Chlorinated Herbicides**

Chlorinated herbicide analytical data will undergo evaluation of the following sample specific criteria:

- C Holding Times
- C Surrogate Recovery
- C Matrix Spike/Matrix Spike Duplicates
- C Laboratory and Field Blanks
- C Target Compound Identification
- C Percent Solids of Sediments

1. Holding Times - Sample data for water, soil, and sediment samples will be evaluated for compliance with holding time criteria provided in Tables 4-1 and 4-2. If holding times are exceeded, flag all data as estimated (~~UU~~for detects, ~~UUJ~~for non-detects). If holding times were grossly exceeded (i.e., more than 2X the holding time), flag all positive data as estimated and reject all non-detects as unusable (~~RU~~).
2. Surrogate Compounds - Analyzed with each sample, blank, and spike unless it is diluted out. Recoveries will be reviewed against limits specified in Sections 3.3.4 and 3.3.5 of this QAPP. For chlorinated herbicides, the recommended surrogate compound is 2,4-dichlorophenylacetic acid. No qualification is done if surrogates are diluted beyond detection. If the surrogate recovery is greater than the upper acceptance limit and greater than 120%, flag all associated positive data as estimated and do not qualify non-detects. If the surrogate recovery is below the lower acceptance limit but greater than 10 percent, flag all associated results as estimated (~~UU~~for detects, ~~UUJ~~for non-detects). If a surrogate has a recovery less than 10 percent, flag all associated positive data as estimated and reject all non-detects (~~RU~~) as unusable.
3. Matrix Spike/Matrix Spike Duplicate Analysis - Spike recoveries and relative percent differences (RPDs) will be reviewed against the limits provided in

Section 3.3.4 and 3.3.5 of this QAPP. Do not qualify associated sample results on the basis of the MS/MSD data alone. Use the MS/MSD results in conjunction with other QC criteria to determine the need for qualification of associated data. If the MS and MSD both have less than 10 percent recovery for an analyte, reject non-detect results for that analyte and qualify positive results for that analyte as estimated for the sample used for the MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

4. Blanks - Laboratory blank data and field blank data will be evaluated for contamination. If any blank has a positive result, qualify associated results as follows:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as a non-detect (UL). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detected at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

5. Compound Identification - All herbicides identified in the primary analysis will be evaluated to determine whether the results are confirmed on a second column. Reject all positive results identified on the quantitation column but not confirmed on a second column. The retention times of sample compounds must be within the calculated retention time windows for both quantitation and confirmation analyses. Reject (R) all positive results not

meeting retention time window criteria unless associated standard compounds are similarly biased. The identification for one or more detected target analytes for ten percent of the sample data will be checked back to the raw data.

- Percent Solids of Sediments - Qualify as estimated all the results of a sample that have percent solids between 10%-50% (i.e., moisture content between 50%-90%) that were not previously qualified as estimated due to other QC criteria. Reject all the results of a sample that has percent solids less than 10% (i.e., moisture content greater than 90%) that were not previously rejected due to other QC criteria.

### **8.2.2.3 Total Extractable Petroleum Hydrocarbons**

Total extractable petroleum hydrocarbon (TEPH) analytical data will undergo evaluation of the following sample specific criteria:

- C Holding Times
- C Matrix Spike/Matrix Spike Duplicates
- C Laboratory and Field Blanks

1. Holding Times - Sample data for water, soil, and sediment samples will be evaluated for compliance with holding time criteria provided in Tables 4-1 and 4-2. If holding times are exceeded, flag all data as estimated (U) for detects, (UU) for non-detects. If holding times were grossly exceeded (i.e., more than 2X the holding time), flag all positive data as estimated and reject all non-detects as unusable (RU).

2. Matrix Spike/Matrix Spike Duplicate Analysis - Spike recoveries and relative percent differences (RPDs) will be reviewed against the limits specified in Section 3.3.8 of this QAPP. Do not qualify associated sample results on the basis of the MS/MSD data alone. Use the MS/MSD results in conjunction with other QC criteria to determine the need for qualification of associated data. If the MS and MSD both have less than 10 percent recovery for an analyte, reject non-detect results for that analyte and qualify positive results for that analyte as estimated for the sample used for the MS/MSD analysis. If the MS and MSD both have greater than 200 percent recovery for an analyte, reject detected results for that analyte and qualify non-detect results for that analyte as estimated for the sample used for the MS/MSD analysis. Use professional judgement in applying this criterion to other samples.
3. Blanks - Laboratory blank data and field blank data will be evaluated for contamination. If any blank has a positive result, qualify associated results as follows. If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag sample result as a non-detect (U). If the sample result is reported as detected at a concentration less than the reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detectable at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

#### **8.2.2.4 Radiochemical Data ( $^{210}\text{Pb}$ , $^{137}\text{Cs}$ , $^7\text{Be}$ )**

1. Recovery Factors: For the lead-210 analyses, check that the chemical recovery or yield is greater than 20 percent. Reject all associated data if recovery is less than 20 percent.
2. Reagent blank data for lead-210 analyses (one per analytical batch). A review of blank data will include identification of detector used for counting, date and time analyzed, number of samples in the analytical batch that the blank is included in, type of blank used, and detection level (as determined by long term background counts) shall be included. Acceptable blank results should have activities less than the detection limit.
3. Lab Replicate Analyses: Duplicate analyses (i.e., duplicate counts) on same mount for one per 20 field samples analyzed by each system. Data on precision will include detector used, analyst's initials, date analyzed, sample ID, value obtained for sample, value obtained for replicate analysis, mean value, duration of counts in minutes for samples and background, number of CPMs for samples and background. Allowable limits are overlap of the 2F precision bands. Qualify associated data as estimated if the above criterion is not met.

#### **8.2.2.5 Other Analytes**

Data validation will consist of the applicable portions of the following as defined by QA/QC sample specifications contained in this QAPP and the analytical methodology:

- C Evaluation of compliance to holding time limits, with data outside of the holding time limits specified in Tables 4-1 and 4-2A qualified as estimated (or



rejected if in the professional judgement of the validator the data are unusable).

- C Evaluation of spike recoveries (matrix spikes) and duplicate analysis precision (field duplicates, matrix spike duplicates) with data outside of the accuracy and precision limits specified in Section 3.2.9 qualified as estimated, ~~UU~~(or rejected, ~~UR~~, if in the professional judgment of the validator the data are unusable).
- C Evaluation of field blank contamination with validation of data from samples associated with contaminated blanks using the following guidance.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. No positive sample results should be reported unless the concentration of the analyte in the sample exceeds five times the amount (in any blank). In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value. Specific actions are as follows:

1. If a compound is found in a blank, but not found in the sample, no action is taken.
2. If a blank has a positive result for an analyte, qualify associated sample data as follow:

If the sample result is greater than the laboratory reporting limit but less than 5 times the blank concentration, flag the sample result as a non-detect (~~UU~~). If the sample result is reported as detected at a concentration less than the

reporting limit and less than 5 times the blank concentration, qualify the sample result as not-detected at the laboratory reporting limit. For aqueous blanks applied to soil/sediment samples, compare the sample result to the equivalent concentration of the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the sample aliquot analyzed.

The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5 x criteria, such that a comparison of the total contamination is actually made.